

# Methodological problems with the classification and measurement of soils containing carbonates

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## Abstract

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Soils containing carbonates are common and are found in various regions around the world. Carbonate content in soils varies and ranges from several to tens of percent. It mainly depends on the nature of the parent rock and the soil-forming processes to which the soil was/is subjected. In some cases, soil carbonate content is affected by an admixture of allochthonous material (anthropogenic and/or natural). Soils containing carbonates can often be problematic both in terms of their classification and analysis. When classifying such soils, it is important to distinguish between primary and secondary carbonates and to take into account the presence of carbonate skeletal parts. The determination of soil carbonate content depends on its nature – taking this into account, analytical procedures may differ depending on local soil chemistry. Carbonates, due to interactions between individual chemical substances, can generate a variety of methodological problems when determining soil characteristics. This fact applies to physical properties such as particle size distribution, chemical and adsorption properties (e.g. content of soluble phosphorus, cation exchange capacity), but also to the determination of the examined micromorphological characteristics. Carbonates also often affect the results of organic carbon analysis, especially in light of the development of modern measurement techniques. This paper attempts to look at common problems related to the systematics and analysis of soils containing carbonates, and presents the solutions used. We believe this paper will be helpful to those who work with carbonate soils.

## 1. Introduction

The term “carbonate soils,” although relatively commonly used by soil scientists around the world, does not have a standardized qualitative and quantitative definition. It is often used interchangeably with “calcareous soils,” although it seems to cover not only limestone soils, but all soils containing carbonates, the origin of which may vary. Some authors, however, frequently use the term “calcium carbonate-rich soils” (Kowalska et al., 2017, 2019) and this refers to soils developed on

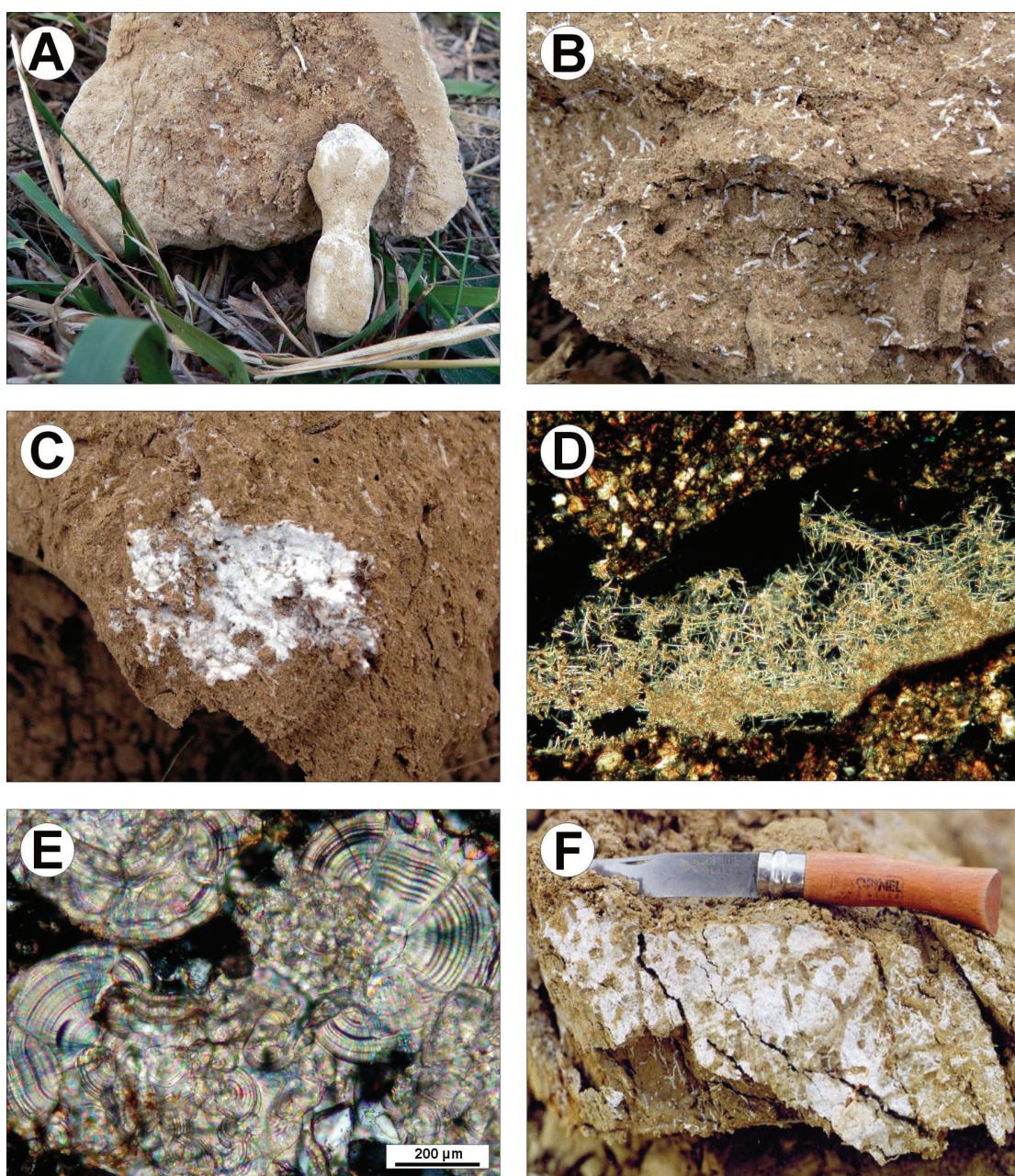
parent material with a calcium carbonate content of 5% to 50% (Czermiński, 1955; Kowalska et al., 2019).

Soil containing carbonates are found all over the world (Zamanian et al., 2016a). The carbonate content in solum may vary between 1% and more than 90% (Razzaghi et al., 2021). Their form may also vary. In the most basic sense, carbonates may be grouped as either primary (lithogenic) or secondary (pedogenic). Lithogenic carbonates, also called geogenic carbonates, are derived from the parent material of the soil – weathered carbonate rock such as limestone, marl, gaize, dolomite, etc. (Guo et al.,

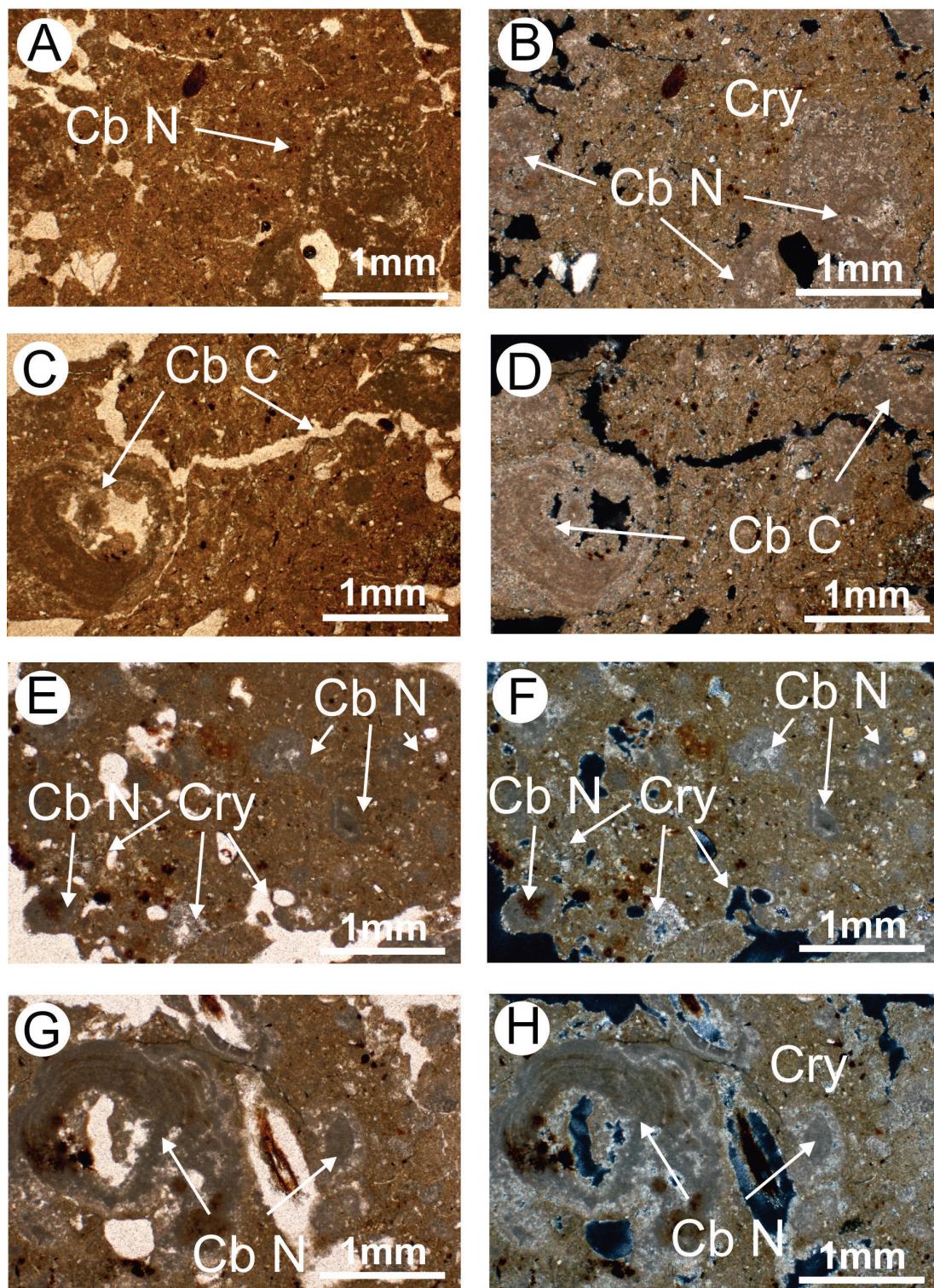
2016). They can also be allochthonous in nature and appear in soil by sedimentation from other locations with calcareous dust or as a result of mass movements of earth (West et al., 1988; Zamanian et al., 2016a; Kacprzak et al., 2006). Selected lithogenic carbonates may also be of zoogenic origin, and their accumulation may take place in either wet or shallow-water environments – e.g. in mollusk shells (Zamanian et al., 2016b). On the other hand, pedogenic carbonates are associated with soil-forming processes taking place in the soil, including the dissolution of carbonates and their subsequent precipitation from the soil solution (Landi et al., 2004), usually following displacement in the soil profile. They can take various morphological forms, such as pseudomycelia, nodules, pebbles, puppets, or create specific soil horizons – Petrocalcic (Buglio et al., 2017; Durand et al., 2010; West et al., 1988). Secondary carbonates also include compounds

of anthropogenic origin, supplied to the soil as a result of human activity, and may assume the form of an admixture in natural soil, or the anthropogenic parent rock from which the given soil is derived may contain carbonates and is usually rich in them.

Carbonates may occur in soil in various physical forms, which is conditioned by their origin and also subsequent conversions (Batjes, 1996; Zamanian et al., 2016a). Secondary carbonates precipitate from solution in the form of nodules, of various size, from fractions of a millimeter to several or even tens of millimeters (Kowalska et al., 2020a). They may also coat soil grains. Examples of secondary forms of carbonate obtained from the soils of the Proszowski Plateau are shown in Figure 1. It should be added here, that carbonate nodules fragmented as a result of hillslope processes were found in soils classified as Cambisols, Calcisols or Regosols in Carpathian Mts. (Fig. 2).



**Fig. 1.** Secondary carbonates occurrence found in chernozems of the Proszowski Plateau (South Poland): A) cemented calcareous nodule, B) rhizolites, macroscopic view on the broken loess block, C) pseudomycelium, macroscopic view on the broken loess block, D) pseudomycelium, microscopic image (XPL, crossed polarizers), E) biospheroids, microscopic image (XPL, crossed polarizers), F) soft carbonate coatings on a soil aggregate surface (based on Żyła, 2007)



**Fig. 2.** The secondary forms of carbonates in soils developed on calcium carbonate-rich parent material at the area of Carpathians Mts. Explanations:

(A, B): Cb N – calcium carbonate nodules (concentric);

Cry – crystallitic b-fabric; (C, D): Cb C – channel calcitic hypocoatings;

(E, F): Cb N – typic carbonate nodules, Cry – crystallitic b-fabric;

(G, H): Cb N – geodic carbonate nodules. Bar length = 1 mm.

A, C, E, G – PPL microphotographs;

B, D, F, H – XPL microphotographs (based on Kowalska et al., 2020a, 2020b)

According to the Polish Soil Classification (2019) (PSC, 2019), soils developed from calcium carbonate-rich parent material may be classified via at least five orders.

First, Order 1: Poorly developed soils: Type 1.1 Raw mineral soils (i.e. raw rocky rendzinas, raw debris rendzinas), Type 1.3: Ordinary rendzinas where all subtypes are characterized by the presence of calcium carbonate in the parent material horizon. Following the principles of PSC 2019, the calcium car-

bonate-rich soils may also belong to Order 2: Brown earths, where Type 2.1, Brown soils (ordinary brown soils, leached brown soils, and humic brown soils) and Type 2.2. Brown rendzinas (all subtypes) are distinguished. Further, Order 4: Clay-illuvial soils, Type 4.1. Clay illuvial soils (typical clay illuvial soils, eroded clay illuvial soils, texturally-contrasted clay illuvial soils, humic clay illuvial soils and vertic clay-illuvial soils) could be considered in the process of classification. Finally,

Order 5: Black soils including some soil types which can be enriched in calcium carbonate, such as: Type 5.1. Chernozems, Type 5.2. Black earths as well as Type 5.3. Chernozemic rendzinas (all subtypes). Finally, carbonates may be found in soils belonging to Organic soils, Type 8.1 Peat soils (gyttja peat soils), Type 8.2 Limnic soils (gyttja soils), Type 8.3 Murshic soils (gyttja murshic soils) and Type 8.4 Folisols (calcareous folisols).

In PSC 2019 the group of soil limnic materials also includes Quaternary limnic carbonate deposits – organic or mineral materials formed in the aquatic environment by precipitation of carbonate from solution as well as the deposition and processing of the remains of organisms containing carbonates and deposition of transferred organic matter. Carbonate limnic sediments may be found in muck soils (OM), peat soils (OT), limnic soils (OJ), gley soils (GG), proper rendzinas (SR), and proper deluvial (SL) soils.

The World Reference Base for Soil Resources (IUSS Working Group WRB, 2015), like many national taxonomies, allows the characterization of soils containing carbonates – calcium carbonate-rich soils can be classified very differently based on diagnostic horizons, diagnostic properties, and diagnostic materials. From the WRB 2015 point of view, Calcisolso consist of the main reference group of soils, where the accumulation of calcium carbonate of pedogenic origin must occur. Nevertheless, in accordance with WRB 2015 principles, calcium carbonate soils may be classified as another reference group, if only a given group allows adding one of the principal and supplementary qualifiers, such as: calcic qualifiers (the horizon, where the accumulation of secondary calcium carbonate occurs), and calcaric qualifiers (the presence of primary carbonate material), respectively. When analyzing the key for the WRB classification, its 25 reference groups can have at least one carbonate type. Only six Reference Soil Groups containing acidic soils – Podzols, Plinthosols, Nitisols, Ferrasols, Acrisols, and Alisols – cannot have any principal and supplementary carbonate qualifiers. As the present paper concerns all soils, the term “soils containing carbonates” will be used hereafter.

A concept related to carbonate soils is active calcium carbonate, defined as a pedogenic, mobile form of  $\text{CaCO}_3$  that is easily soluble in soil water (Gras, 1972, Polish Soil Classification, 1989; Srivastava et al., 2002). An excess of active carbonate causes a decrease in the amount of humus compounds as well as deterioration of their quality by hindering polymerization of multiparticle humus bonds (Kuźnicki and Skłodowski, 1976; Kowaliński and Licznar, 1986). The active form of calcium carbonate is, however, typical of highly calcareous soils in arid and semi-arid climates. Nonetheless, estimation of the quantity of mobile calcium carbonate does not include the total amount of  $\text{CaCO}_3$  in the soil solution, since some amount is adsorbed by plant roots at any given moment (Gras, 1972). Moreover, calcium carbonate displacement such as drainage, precipitation, and evaporation does not allow for a complete evaluation of mobile calcium carbonate amounts (Gras, 1972). Thus, the main source of active calcium carbonate should exist in the solid soil state. Active calcium carbonate is, however, a rather complex and controversial entity in soil science and should be discussed in detail. Due to unique chemical and

physical features, soils containing carbonates are problematic in laboratory analysis. The problem with the analysis of soils containing carbonates has been investigated for a long time (e.g. Miklaszewski, 1930; Yaalon, 1957; Dobrzański, 1987; Kuźnicki and Skłodowski, 1975; Duchaufour 1976). Furthermore, from many discussions and experiences, we are aware of the uncertainty associated with soils containing carbonate, in terms of soil classification, chemical and physical properties assessment, and micromorphological research. Therefore, the following question arises: Should soils containing carbonates be analyzed using special procedures? We attempt in this study to assess the main difficulties associated with soils containing carbonates and make an effort to yield a comprehensive summary of issues to be considered. Hence, the purpose of this work is to identify certain difficulties, and interpret research results in the area of the quantitative analysis of soils containing carbonates.

## 2. Soil description and soil classification

The first issue is the problem with the classification of soil containing carbonates. There are different aspects in the WRB (IUSS Working Group, 2015) and PSC (2019) that introduce many inconsistencies during the classification process.

Most soil description systems consider only the presence of pedogenic carbonates. For example, according to the WRB (IUSS Working Group, 2015; Jahn et al., 2006), the letter ‘k’ denotes the presence of pedogenic (secondary) carbonates. This designation provides insight into soil formation history, and informs about the presence of carbonates. However, in areas with a humid climate (‘udic’, ‘perudic’ soil moisture regime), carbonates are constantly leached out of the soil system; therefore, there are no stable conditions for accumulation of pedogenic carbonates. The general lack of secondary carbonates in soil in such areas makes the presence of primary (lithogenic) carbonates very important due to the strong effect of carbonates on soil properties. However, the WRB (IUSS Working Group, 2015) does not allow to indicate primary carbonates, and the presence of primary forms is omitted in the course of soil description.

A good solution to this problem may be found in PSC (2019). This system uses the suffix ‘ca’ for subordinate distinction, which emphasizes specific characteristics of the parent material (presence of lithogenic carbonates). For example, in certain soils found in Poland, calcareous rock fragments occur in non-calcareous fine earths in the pedon (PSC, 2019). The presence of these rock fragments makes a soil at least neutral, whereby it would be acidic in udic conditions.

The problem with the systematic description of soils containing carbonates may appear in humid regions, where intensive agricultural activity has been performed for many centuries. As a result of the erosive flattening (truncation) of the profiles of lessive (clay-illuvial) soils and chernozems, loose calcareous parent material may be present directly below the plowed horizon. However, these are not weathered, carbonate rocks, but pedogenic  $\text{CaCO}_3$  concretions. Soils with such a mor-

phology have been often described both in the landscape of young glacial moraine uplands (Podłasiński, 2013; Świtoniak, 2014; Matecka and Świtoniak, 2020) as well as in loess areas (Klimowicz and Uziak, 2001; Drewnik and Żyła, 2019; Woźnica et al., 2019; Źižala et al., 2019; Jenčo et al., 2020). A significant amount of carbonate concretions in these soils results from the illuvial accumulation of secondary carbonates in the base of well-formed soils (chernozems or lessive soils) prior to the erosion stage. As a result of agriculturally-induced soil erosion, the horizon containing carbonate nodules became the topsoil horizon. However, following the rules of the WRB (IUSS Working Group, 2015), the presence of these nodules should be marked with the letter 'k.' In fact, these nodules are not actually formed (as pedogenic carbonates), as they dissolve in the process. They are generally equivalent to gravel or stones with some calcium cement (Drewnik and Żyła, 2019). Therefore, adding the letter 'k' leads to a misunderstanding. The presence of carbonate nodules within the topsoil horizon, which had formed under different soil formation conditions (prior to the period of intensive agriculture), should be marked with a symbol appropriate for lithogenic carbonates. In fact, at present, they serve as components of parent material affecting present-day soil properties. The introduction of the suffix 'ca' in the WRB 2015 description (or one-letter suffix in accordance with WRB rules), similarly as in the case of PSC 2019, seems to be a good solution.

Yet another problem with the classification of soils containing carbonates in skeleton particles (> 2 mm) may be described in terms of typology. For example, according to PSC (2019), poorly developed soils containing more than 10% (weight average) carbonate nodules in the total weight of skeleton particles in the 30–60 cm layer should be classified as "proper pararendzinas." Failure to meet this key criterion makes it necessary to classify these soils as regosols, where the presence of calcium carbonate may be identified only at the variant level (carbonate regosols). With soils developed from loess, their classification as pararendzinas does not raise any doubts due to the fine texture of the parent rock, in which the main section of the skeleton consists of carbonate concretions (Drewnik and Żyła, 2019). Concerns may be raised mainly in the case of soils made of boulder clays, in which the proportion of the non-carbonate skeleton and carbonate concretions substantially varies from site to site and is quite difficult to estimate in the course of fieldwork (Matecka and Świtoniak, 2020). Therefore, it is necessary to develop a methodology to determine the share of the carbonate skeleton in the total mass of the given skeleton.

### 3. Carbonate content test based on CO<sub>2</sub> volume measurements

In a laboratory determination of carbonate content, the following data may be evaluated: volume of released CO<sub>2</sub> (FAO, 2020), pressure induced by released CO<sub>2</sub> (Burt, 2004), chemical effect caused by released CO<sub>2</sub> (van Reeuwijk, 2002). Whichever of the above methods is used, the mass of CO<sub>2</sub> released is measured directly. This gas may be the product of decomposition

of various possible carbonates; therefore, in fact the final result of laboratory measurement is only the percentage of CO<sub>2</sub> released from soil by weight. In many cases, following laboratory measurement, a stoichiometric conversion is used to obtain a percentage by weight of CaCO<sub>3</sub>, as calcite is the most commonly found carbonate mineral in soil (Lal et al., 1999). However, in many cases, other carbonate minerals may occur in soil (magnesian calcites, dolomite, siderite in hydromorphic soils, etc...), and such a stoichiometric conversion may be burdened with an error that is difficult to estimate.

Only the notation "the percentage by weight of CO<sub>2</sub> released from the soil" should be used in databases. After calculations carried out on the basis of the volume of CO<sub>2</sub>, the prefix "eq" (as in "eqCaCO<sub>3</sub>") should be provided to emphasize that this is merely the equivalent of CaCO<sub>3</sub>. This rule is used in many studies, but not always, which can lead to misunderstandings.

### 4. Determination of (removal of) carbonates in soils

The most frequently used determination method for carbonate content in the soil, based on carbonate decomposition with hydrochloric acid (EN ISO 10693:2014), also causes other kinds of problems. As mentioned above, the volume of released CO<sub>2</sub> is readily measured and the method is considered simple. However, when analyzing carbonate content in soil, it should be remembered that apart from calcium carbonate, soils also contain, as mentioned above, other carbonates – magnesium and iron, and magnesium and calcium carbonate. In the course of the analysis with the method based on the neutralization of carbonate with hydrochloric acid, the other carbonates decompose much more slowly. Failure to take this fact into account may lead to errors that are difficult to estimate. While the rate of decomposition of calcium carbonate in an analysis of soil containing an average amount of carbonate is estimated to be merely 1 to 2 minutes, a similar amount of dolomite decomposes in 1 to 2 hours (Loeppert and Suarez, 1996). However, some of the proposed analytical methods assume leaving the analyzed sample until the next day (van Reeuwijk, 2002).

Decomposition/removal of carbonates is required before pursuing other analyses. Failure to remove carbonate from the sample may, for example, lead to an overestimation of the cation exchange capacity (CEC) (e.g. by using ammonium acetate at pH = 7), which represents one of the fundamental properties of soil. Also in this case, the length of time required for its removal through acid fumigation is estimated to be at least several to tens of hours (Harris et al., 2001; Ramnarine et al., 2011). An additional problem also occurs in the presence of iron (II) carbonate, i.e. siderite. Siderite is so poorly soluble that it cannot be detected with a 'standard champagne test'. In this case, Vinduskova et al. (2019) propose a 4-day procedure for the effective elimination of iron(II) carbonate from the soil sample.

In order to avoid the abovementioned problems, a different laboratory procedure may be used including heating the sample, using more concentrated acid, and grinding the sample (Soil Science Division Staff, 2017).

## 5. Measuring the CEC of soils containing carbonates

The measurement of the CEC of carbonate-bearing soil is a problematic endeavor. Ammonium acetate ( $\text{pH} = 7$ ) is a commonly used reagent for the extraction of base cations (Ca, Mg, K, and Na) from the soil adsorption complex (van Reeuwijk, 2002). The CEC value obtained based on ammonium acetate extraction is also commonly used in soil classification systems (e.g. WRB) for classification purposes (e.g. as criteria used in the definition of key qualifiers). However, extraction of base cations using ammonium acetate at  $\text{pH} = 7$  leads to the overestimation of the quantity of these cations. In order to solve this problem, other extraction methods are proposed for carbonate soils (e.g. ammonium chloride buffered at  $\text{pH} = 8.2$ ; sodium acetate buffered at  $\text{pH} 8.2$ ; barium chloride –triethanolamine (TEA) buffered at  $\text{pH} 8.1$ ; and a few others) (Pansu and Gautheyrou, 2006).

## 6. Measuring the content of organic carbon in the presence of carbonates

Soil carbonates may be problematic in terms of soil organic carbon analysis – especially when carbonate content is high. Proper sample preparation procedures provided by equipment manufacturers should be utilized with the dry combustion method of total carbon analysis (EN 15936:2012). Depending on the methodology adopted, the result should be adjusted for the content of inorganic carbon in the sample, or the given sample should be prepared in an appropriate manner, chemically removing the carbonates beforehand (Dhillon et al., 2015).

## 7. Measuring particle size distribution of soils containing carbonates

Particle size distribution (PSD) is one of the most important soil characteristics. This key metric is determined in nearly all soil investigations and affects many soil properties: biological (Wolińska et al., 2011; Hemkemeyer et al., 2018), physical (Lamorski et al., 2013; Widomski et al., 2015; Szypłowska et al., 2019), and chemical (Brogowski and Chojnicki, 2013; Tkaczyk et al., 2017; Waroszewski et al., 2019). There are many methods for PSD determination, but generally speaking only two types of methods are very commonly used: i) sieve-sedimentation methods (SSMs), and ii) the laser diffraction method (LDM) (Biegagnowski et al., 2018; Makó et al., 2019).

SSMs consist of two main stages. The coarser fractions (sand) are measured using a set of sieves. The finer fractions (clay and silt) are measured in the course of the sedimentation process. Next, the most commonly employed SSM procedures involve the use of the pipette and hydrometer methods (Soil Survey Staff 2014; ISO 11277:2020); however, new approaches are also being developed (Kaszubkiewicz et al., 2020).

The presence of carbonates in soils examined via sieve measurements is usually not very significant. Carbonate crumbs behave in exactly the same way as other soil particles/aggregates. The only case in which this measurement may be burdened with

greater uncertainty is when the crumbs possess a fresh, sharp geometry, or more accurately speaking have a needle shape. The studied needles that possess a dimension larger than the nominal sieve mesh are able to pass through when they are positioned perpendicularly to the sieve surface. A much more complex situation is observed with the sedimentation measurement process. The density of solid mineral particles is usually  $2.65 \text{ Mg m}^{-3}$ , while the densities of carbonates are different, depending on the kind of carbonate in question. For instance, chalk density reported by Bell (2005) may be  $1.25 \text{ Mg m}^{-3}$ , while siderite may have a density equal to  $4.0 \text{ Mg m}^{-3}$ . When the density of the studied carbonates differs from that of mineral particles, the sedimentation rate is also going to be different. Consequently, the time intervals calibrated for the investigated texture fraction are not valid, and the resultant PSD is burdened with an error that is difficult to estimate. Another problem with the sedimentation measurement of carbonates is their behavior in water. Normally it is assumed that calcium carbonate is not soluble in water. This assumption is valid for the purpose of PSD measurement. However, with certain types of carbonates, their physical state in water changes. Solid particles become smearable. In other words, one large particle transforms into a suspension (colloid) of many small particles. And this process produces a major effect on the measured value of PSD.

There are different approaches to the measurement of PSD by sieve sedimentation method when carbonates are present in the soil. Carbonate removal is one recommended approach (ISO 11277:2020, Soil Survey Staff, 2014). There are many studies that follow this recommendation (Eshel et al., 2007; Bartkowiak, 2011). However, when the carbonate content is very high its removal prior to PSD measurement does not make any sense (Lemkowska and Sowiński, 2018; Al-Mamoori et al., 2019). The PSD measurement of the entire soil sample (i.e. without carbonate removal) is carried out in such a case (Dobak and Wyrwicki, 2000; Lemkowska and Sowiński, 2008; Asgari Hafshejani and Jafari, 2017; Kowalska et al., 2017).

Use of the LDM for the measurement of PSD in soils in which carbonates occur solves the problem of different density of carbonates (Bartmiński et al., 2011). This is due to the use of a suspension, where the soil is intensively stirred and pumped through the measuring cell. Nonetheless, the problem of the water effect on carbonates and the described above resultant reduction of carbonate particle size remains, especially in active calcium carbonates. However, the error in PSD measurement using LDM is much smaller than the error generated when using sieve-sedimentation. This is because the LDM measurement is much quicker than the use of the SSM method.

## 8. Measuring the content of soluble phosphorus in soils containing carbonates

When determining the extent of human impact on the development of humus horizons, it is important to determine the content of phosphorus soluble in 1% citric acid (PCIT). A value above  $650 \text{ mg PCIT kg}^{-1}$  of soil is considered to be one of the criteria for the identification of the anthropogenic horizon antrik

(PSC, 2019). These horizons very often contain calcium carbonate – for example from fertilizer use, which may cause lowered results in terms of PCIT content. To prevent this, in samples with carbonate content > 0.3% (it is assumed that a lower  $\text{CaCO}_3$  content does not significantly affect the result), an additional portion of powdered citric acid is added prior to extraction – for each additional 0.1% calcium carbonate, 7 mg of citric acid is added (van Reeuwijk, 2002). The use of a standard amount of acid (such as would be used in carbonate-less samples) in the presence of carbonates is not sufficient in order to extract the entire amount of phosphorus. The use of increased amounts of citric acid has been successfully tested, for example, on samples containing carbonates in anthropogenically-altered material at archaeological sites found in the Chełmno region of Poland (Markiewicz, 2012). In material taken from carbonate soils characterized by high pH values, the determination of extractable forms of phosphorus may encounter some problems (Spychalski et al., 2018). In such cases, to get the right results, methods based on the extraction of phosphate from the soil by 0.5 N sodium bicarbonate solution (adjusted to pH 8.5) should be employed. For example, the Olsen Method (Olsen and Sommers, 1982) is used for the determination of bioavailable forms of inorganic ortho-phosphate ( $\text{PO}_4\text{-P}$ ) in soils with pH ranging from neutral to alkaline. The CAL method (Schüller, 1969) is also recommended and widely used for alkaline soils – it is based on the extraction of phosphorus with calcium acetate, calcium lactate, and acetic acid.

## 9. Preparation of thin sections in micromorphology investigations of soils containing carbonates

Micromorphological analyses are performed on soil thin sections. In order to prepare the thin sections, undisturbed and oriented soil samples are required (Srivastava et al., 2009). Then, the soil sample requires impregnation. One of the better known impregnation techniques calls for saturation with a resin (epoxy or polyester), blended with acetone or another solvent that reduces surface tension for better impregnation (FitzPatrick, 1984; Menzies and Meer, 2018; Murphy, 1986). With the use of this method, a resin hardener (catalyst) is combined with the resin and acetone and then the mixture is gradually added to the soil sample. After a “soaking” bath in the resin, samples are left to impregnate for 1–2 days in a vacuum chamber (Srivastava et al., 2009). Then, the samples should be left to harden, which may last from one week to one month, depending on the amount of catalyst and acetone added. Properly impregnated samples are hard, homogeneous, and exhibit high durability and physical resistance (i.e. they do not crack or crumble). This enables them to be cut with a diamond saw and polished.

However, soil samples are not always perfectly saturated and hardened by an epoxy resin as a whole. This may be due to mistakes made during the impregnation process (not using the recommended doses of resin and catalyst, no acetone added, an excessively rapid transition to the next stage of the analysis) or due to soil properties: high content of soil organic matter and soil texture (specifically a high content of the clay fraction)

(Ferreira et al. 2016). The problem with improper impregnation with resin may be especially troublesome in the case of carbonate soils due to the pattern of weathering of their parent material: e.g. limestone, which favors clay fraction formation (Konecka-Betley, 1976; Menzies and Meer, 2018). Additionally, the texture of carbonate soils is also determined by the deposition of allochthonous material on the soil surface that is often rich in silt or sand particles. As a result, the particle size distribution of carbonate soils is characterized by high content of clay or silt featuring a fairly low degree of permeability (Konecka-Betley, 1976). In the context of micromorphological research, such a “heavy” nature of the texture may hinder the penetration of the soil sample by the resin (Ferreira et al., 2016). As a result, undisturbed calcium carbonate-rich samples undergoing impregnation can become at least partly or completely unsaturated by the resin, and not fully hardened, and this may contribute to the emergence of certain problems (Menzies and Meer, 2018) such as (1) difficulties in the cutting of not fully hardened soil that falls apart during cutting, (2) the sample cannot be polished before placing it under the microscope, as it is too soft; 3) part of the soil sample detaching from the thin section during lapping; 4) loss of individual soil elements from the sample may occur during subsequent stages of thin section preparation.

Another problem is the impregnation of carbonate pedofeatures. The secondary forms of calcium carbonate develop due to pedogenic processes such as leaching and recrystallization, and sometimes may form so-called “hard carbonate nodules” (Kovda and Mermut, 2010). This may cause some difficulties during epoxy resin impregnation. Failure of impregnation in such nodules may result in their loss in the preparation of thin sections or their defragmentation.

Another issue related to the impregnation of carbonate-rich soils with some epoxy resins (e.g. Araldite® 2020) is that the resin boils too quickly (within one hour) during impregnation under vacuum. The heat released during boiling accelerates resin hardening, and may affect the microscopic features of minerals containing water in their structure.

There exist several different solutions to avoid problems with impregnation. First, it is recommended to remove soil moisture in acetone vapor in the course of drying clay-rich, wet samples. The method involves placing the soil samples in a chamber with acetone vapor and leaving them for a few weeks. Subsequently, the water mixed with excess acetone is displaced and removed from the soil samples. This process should be repeated up to 6 times to be sure that all water is removed from the sample (Murphy, 1985; Srivastava et al., 2009). Second, after saturation with epoxy resin, the soil samples may stay longer in the vacuum chamber (1–2 weeks) so that the resin can reach even the smallest pores due to infiltration via capillary action. Third, more acetone (up to 15%) may be added to the mixture of epoxy resin and catalyst. As a result, the resin is diluted, which enables impregnation of the soil sample by the epoxy resin. Another way to impregnate clay-rich carbonate soil samples consists of the addition of epoxy resin mixed with acetone at set intervals – about 3 ml every ten minutes (if the soil sample is collected in a Kubiena box). The soil samples should remain in a vacuum chamber. The epoxy resin mixed

with acetone should be applied until it seeps into the soil – also through capillary action. This may take up to a few hours. An alternative to the above-mentioned methods is the use of a different kind of resin – a polyester resin designed for clayey-rich soil (Sohaib et al., 2018).

## 10. Conclusions

Many standard methods used in soil measurement are sensitive to carbonates. The best solution is the removal of carbonates in such cases. However, when the carbonate content is high or very high, its removal completely changes the sample and only the residue is examined, and not the soil as such. The following question then arises: What are we actually measuring? On the other hand, sometimes the removal of calcium carbonate is not possible (e.g. thin section preparation). The solution to this problem is not so easy, and therefore the standard procedures should be used with the awareness that errors caused by the presence of carbonates may occur. However, a very important issue in such cases is the detailed description of the research method used, with an emphasis on potential inaccuracies resulting from the presence of carbonates.

Regarding soil classification when generating soil descriptions, the introduction of the additional symbol 'ca' indicating the presence of lithogenic carbonates would be useful from a practical point of view.

We do hope that our work will help to address the problems of those working with soils containing carbonates, and that it will be continued by discussing other methods of analysis.

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## Metodologiczne problemy z klasyfikacją i pomiarami w glebach zawierającymi węglany

### Słowa kluczowe

Węglany  
Gleby  
Metody pomiaru gleb  
Klasifikacja gleb  
Metrologia gleb

### Streszczenie

Gleby zawierające węglany są powszechnie i występują w różnych rejonach na całym świecie. Zawartość węglanów w takich glebach różna i waha się od kilku do kilkudziesięciu procent. Zależy przede wszystkim od charakteru skały macierzystej oraz procesów glebotwórczych, którym dana gleba podlegała / podlega. W niektórych przypadkach na zawartość węglanów wpływa domieszka materiału allochtonicznego (antropogenicznego i / lub naturalnego). Gleby zawierające węglany często mogą być problematyczne zarówno w zakresie ich klasyfikacji jak również analizy. Przy klasyfikowaniu gleb zawierających węglany ważne jest rozróżnienie między węglanami pierwotnymi i wtórnymi oraz uwzględnienie obecności węglanowych części szkieletowych. Oznaczenie zawartości węglanów w glebie zależy od ich charakteru – biorąc to pod uwagę poszczególne procedury analityczne mogą różnić się w zależności od ich składu chemicznego. Węglany, ze względu na interakcje między poszczególnymi substancjami, mogą generować problemy metodologiczne przy określaniu wybranych cech gleb. Dotyczy to właściwości fizycznych, takich jak skład granulometryczny, ale także określania cech mikromorfologicznych. Węglany mają również wpływ na wyniki analizy węgla organicznego, zwłaszcza w świetle rozwoju nowoczesnych technik pomiarowych. W niniejszej pracy podjęto próbę zebrania powszechnych problemów związanych z systematyką i analizą gleb zawierających węglany oraz zaprezentowano zastosowane rozwiązania. Wierzymy, że ten artykuł będzie pomocny dla tych osób, które zajmują się glebami węglanowymi.